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EMPIRICAL CORRECTIONS FOR VARIABLE ABSORPTION OF
SOFT X-RAYS BY MYLAR

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EMPIRICAL CORRECTIONS FOR VARIABLE ABSORPTION OF
SOFT X-RAYS BY MYLAR

by

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Department of the Army Project No. 517-06-002
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ABSTRACT

The characteristic X-rays of many light elements are strongly attenuated by 0.00025-inch Mylar.¹ An empirical method was developed to correct for the effects of variable 0.00025-inch Mylar thickness on the analyses of these light elements. Linear relationships were established between correction factors for aluminum K α radiation and those for sulfur K α and chlorine K α radiations. Aluminum K α correction factors for variable Mylar thickness are determined by measuring aluminum K α intensities transmitted by Mylar samples of interest, and calculating the correction factors directly. The aluminum K α radiations originate from interchangeable aluminum standards. The correction factors for sulfur K α and chlorine K α are determined from the aluminum K α factors and equations of the linear relationships mentioned. The maximum intensity ratios which have been noted for analysis of the same or identical samples against different Mylar films selected at random are: aluminum K α -1.210, sulfur K α -1.064, and chlorine K α -1.038.

¹Polyester film manufactured by E. I. duPont de Nemours and Company, Inc.; Film Department, Wilmington, Delaware.

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EMPIRICAL CORRECTIONS FOR VARIABLE ABSORPTION OF SOFT X-RAYS BY MYLAR

I. INTRODUCTION

Thin Mylar films are used extensively in X-ray fluorescence analysis as supports for samples, and as windows to transmit characteristic X-rays from the samples. The use of Mylar in conjunction with an inverted X-ray geometry has greatly facilitated sample preparation and analyses of a variety of materials. With an inverted geometry the direct analyses of liquids and most slurries must be accomplished on samples placed against Mylar. Although powders can be briquetted, they are frequently placed against Mylar to increase the speed of analysis. With the conventional X-ray geometry (top sample-surface irradiation) the direct analyses of liquids and slurries can be conveniently performed without using Mylar. However, unless an internal standard is used, the precision and accuracy of these analyses is generally less than for analyses performed on samples placed against Mylar. This is particularly true for certain slurries where the composition of an open sample surface may continuously change upon standing.

A primary objection to using Mylar as windows in X-ray fluorescence analysis is that Mylar strongly absorbs characteristic X-rays from many light elements. Table I shows the approximate percentage transmission through nominal 0.00025-inch Mylar of characteristic $K\alpha$ radiations from four light elements commonly analyzed in this laboratory. The percentage transmission differs markedly as the wavelength varies from iron $K\alpha$ (1.94 angstroms) to aluminum $K\alpha$ (8.34 angstroms). The percentage transmission depends on the thickness of the Mylar film and the wavelength of the radiation under consideration.

Another more serious objection to using Mylar as windows is that the thickness of commercially available 0.00025-inch film varies about the nominal value. These variations are sufficiently large to cause significant errors in the analyses of light elements whose characteristic radiations are strongly attenuated by Mylar. A practical, effective method is described here, whereby corrections are made for the effect of 0.00025-inch Mylar thickness variations on the measured intensities of aluminum, sulfur, and chlorine $K\alpha$ radiations. The method should also be applicable to other light elements.

II. INSTRUMENTATION

All determinations were made using a Philips Universal Vacuum X-ray Spectrograph with associated electronics capable of handling high counting rates. Specific instrumentation and operating conditions used for obtaining and applying Mylar correction factors follow:

X-ray tube	Philips FA-60 with tungsten target
X-ray tube setting	50 kilovolts (peak); 45 milliamperes
Entrance collimator	Parallel plates with 0.02-inch spacings
Detector	Gas flow proportional counter
Detector flow-gas	P-10 (90% Argon, 10% methane); 0.5 cu ft/hr flow rate (air calibration)
X-ray optics	Flat-crystal inverted
X-ray optical path	Helium; 1.0 liter/min flow rate (air calibration)
Analyzing crystals	Sodium chloride and Ethylene diamine D-tartrate (EDDT)

III. ALUMINUM STANDARDS

The correction factor method is based on utilizing interchangeable aluminum standards. Consequently, they should be very carefully prepared and handled. Four aluminum standards were prepared, one for each position of the Vacuum X-ray Spectrograph. Adjacent sections were cut from a single piece of 1100-aluminum bar stock. Each section was then machined to fit the standard circular holder of the Vacuum Spectrograph (1½ inches in diameter by 1 inch in length) and to give flat, smooth end surfaces. Other types of aluminum can be used to make the standards.

It is highly desirable that the standards be interchangeable. Otherwise, corrections must be made for aluminum K α intensity differences among standards, and a given standard must always be associated with a particular Mylar sample. Also, the aluminum must contain no impurities which would interfere with the measurement of aluminum K α intensities. The aluminum K α intensities from the standards used in this work differ less than 0.3 percent relative at the 95% confidence level.

The standards are stored in a desiccator when they are not being used. The analyzed surfaces are wiped with a lintless cloth or soft paper towel after analyses are completed for a given day. Aluminum K α intensities from the standards treated in this manner have remained unchanged for the past two years.

IV. MYLAR CORRECTION CURVES

Correction curves for nominal 0.00025-inch Mylar thickness variations were established for chlorine $K\alpha$ and sulfur $K\alpha$ radiations. These curves are shown in Figure 1.

Pertinent analytical parameters are shown in Table II. A fixed count technique was used throughout; all intensity measurements were recorded as the number of seconds required to collect the preselected total counts listed in the table. This eliminated the additional computation to convert to counts per second. Sulfur $K\alpha$ and chlorine $K\alpha$ intensities were measured from a stable pellet containing high concentrations of the elements, and aluminum $K\alpha$ intensities were measured from the aluminum standards. The peak-to-background ratios for sulfur and aluminum were increased with pulse height analysis. Only peak intensities were measured. Since in every instance the peak-to-background ratio exceeded 100 to 1, errors resulting from the uncorrected background component were very small. In general, analytical parameters were chosen to correspond to those employed for subsequent slurry analyses. Since a ratio method was used, the analytical parameters chosen are not critical. Others will probably work equally well.

The method of preparing the Mylar correction curves is illustrated in Table III, where a portion of the actual chlorine $K\alpha$ data are recorded. Similar data were also obtained for preparing the sulfur $K\alpha$ curve. The following procedure is recommended:

A. Load aluminum standards into two sample holders containing nominal 0.00025-inch Mylar films of different thickness. The difference in thickness should be established by prior measurements of aluminum $K\alpha$ intensities transmitted by a larger number of films.

B. Place the loaded sample holders in the two reproducible spectrograph positions. Measure in rapid succession the peak aluminum $K\alpha$ intensities transmitted by the two Mylar samples. Calculate two aluminum $K\alpha$ ratios from the measurements (see Table III for samples 1 and 2).

C. Similarly, analyze a stable pellet against the same two Mylar samples for other peak characteristic radiations of interest (in this example chlorine $K\alpha$). Calculate two corresponding ratios.

D. Analyze the aluminum standards and the stable pellet against additional pairs of Mylar samples, as shown in Table III, to adequately cover the range of Mylar thickness differences encountered.

E. Plot the aluminum ratios against those of the other elements, and fit a straight line to the data of each element. (See Figure 1.)

The equations for the chlorine $K\alpha$ and sulfur $K\alpha$ curves shown in Figure 1 are:

$$C_{Cl} = 0.1780 C_{Al} + 0.8225 \quad (1)$$

$$C_S = 0.3070 C_{Al} + 0.6927 \quad (2)$$

Although the equations were derived in connection with a special type of slurry analysis, it is likely that they can be applied elsewhere. The fact that only peak intensity measurements were made is the main limitation to consider.

The curves in Figure 1 show the magnitudes of errors to expect from duplicate analyses when no corrections are made for Mylar thickness differences. The maximum experimentally determined correction factors in Figure 1 are: aluminum $K\alpha$ -1.210, sulfur $K\alpha$ -1.064, and chlorine $K\alpha$ -1.038. The maximum factors are those found from the analyses of several hundred Mylar samples. Whether analyses of light elements can be accomplished without Mylar corrections depends on the elements and the purpose of the analyses, which in turn dictates the tolerable error. Obviously aluminum determinations using 0.00025-inch Mylar are not practical, even semi-quantitatively, unless the corrections are made. On the other hand, 0.00025-inch Mylar thickness variations have very little effect on the precision and accuracy of iron determinations. Consider the transmission data for iron $K\alpha$ radiation in Table I. The maximum error caused by nominal 0.00025-inch Mylar thickness variations on iron determinations would be about 0.5% relative, and rarely will the maximum error occur. Because of the small error, a correction curve was not constructed for iron $K\alpha$ radiation.

The curves for sulfur $K\alpha$ and chlorine $K\alpha$ in Figure 1 serve as a guide for predicting the influence of Mylar thickness variations on the analyses of other elements. To illustrate, a curve for silicon $K\alpha$ would have a steeper slope than that for sulfur $K\alpha$. Moreover, its correction factors would be intermediate between those for sulfur $K\alpha$ and aluminum $K\alpha$. The exact curve for a particular element can be constructed, of course, according to the foregoing procedure.

V. APPLICATION OF MYLAR CORRECTION FACTORS

The method of correcting raw data for Mylar thickness variations is demonstrated in Table IV for an actual analysis of a single slurry batch. A reference standard and three unknown samples were analyzed as a group for the elements: chlorine, sulfur, and aluminum. The analysis was later repeated for another group from the same batch. All raw data in seconds are tabulated in the appropriate analysis column of the table for each element. Mylar correction factors are applied as follows:

A. Attach a 0.00025-inch Mylar film to the reference standard holder, and to one or more unknown holders depending on the number of unknowns which will be analyzed. The Mylar film for the reference standard holder may be repeatedly used until it is no longer serviceable.

B. Number the sample holders and measure in order with a fixed goniometer setting the aluminum $K\alpha$ intensities transmitted by the films (column 2, Table IV). It is desirable to repeat these measurements as shown in the table. The reference standard holder should be placed in a reproducible spectrograph position, and unknown holders in rotatable positions should be rotated.

C. Analyze the reference standard and the unknowns against the same Mylar films as described for the standard aluminum analyses (analysis columns of Table IV).

D. Calculate the correction factors for aluminum $K\alpha$ radiation: Divide the seconds for the aluminum standard in the reference standard holder by the seconds for each aluminum standard in an unknown holder to get individual correction factors (column 9, Table IV).

E. Calculate the correction factors for chlorine $K\alpha$ and sulfur $K\alpha$ radiations: substitute the aluminum $K\alpha$ correction factors in equations 1 and 2 of the text, and calculate the individual correction factor for each unknown sample (columns 3 and 6, Table IV).

F. Multiply the seconds for each raw data analysis by the corresponding correction factor to obtain analyses corrected within a group for Mylar thickness variations (columns 5, 8, and 11, Table IV).

G. Having corrected for Mylar thickness variations within a group of samples analyzed at the same time, divide the seconds for the reference standard by the corrected seconds for each unknown. Average the resulting ratios to obtain the analytical ratio for each element.

H. Alternatively, first calculate ratios using the raw analysis data, and then correct these ratios for Mylar thickness variations by dividing each ratio by the proper correction factor. This correction method will apply to data produced by quality control instruments, such as the Autrometer, which print out intensity ratios.

The reference standard compensates for Mylar variations among groups, and for short-term and long-term instrument changes. The analytical ratio is the proper measure to use for setting up calibration plots and for checking the batch-to-batch reproducibility of a product.

VI. CONCLUSION

The empirical method described here has been used extensively for the past two years and results have been excellent. In fact, it has permitted light element analyses which could not previously be achieved by direct X-ray fluorescence methods.

TABLE I

TRANSMISSION OF SOFT X-RAYS BY NOMINAL 0.00025-INCH MYLAR

Element	Emission Line	Wavelength, Angstroms	Percent Transmission
Iron	K α	1.94	98.0
Chlorine	K α	4.73	76.1
Sulfur	K α	5.37	68.0
Aluminum	K α	8.34	24.2

TABLE II

ANALYTICAL PARAMETERS FOR DETERMINING MYLAR CORRECTION CURVES

Element	Analytical Line	Peak Angle, Degrees 2 θ	Total Counts Collected	Analyzing Crystal	Pulse Height Analyzer
Chlorine	K α	113.96	1,024,000	NaCl	Integral
Sulfur	K α	144.78	64,000	NaCl	Differential
Aluminum	K α	142.74	128,000	EDDT	Differential

TABLE III

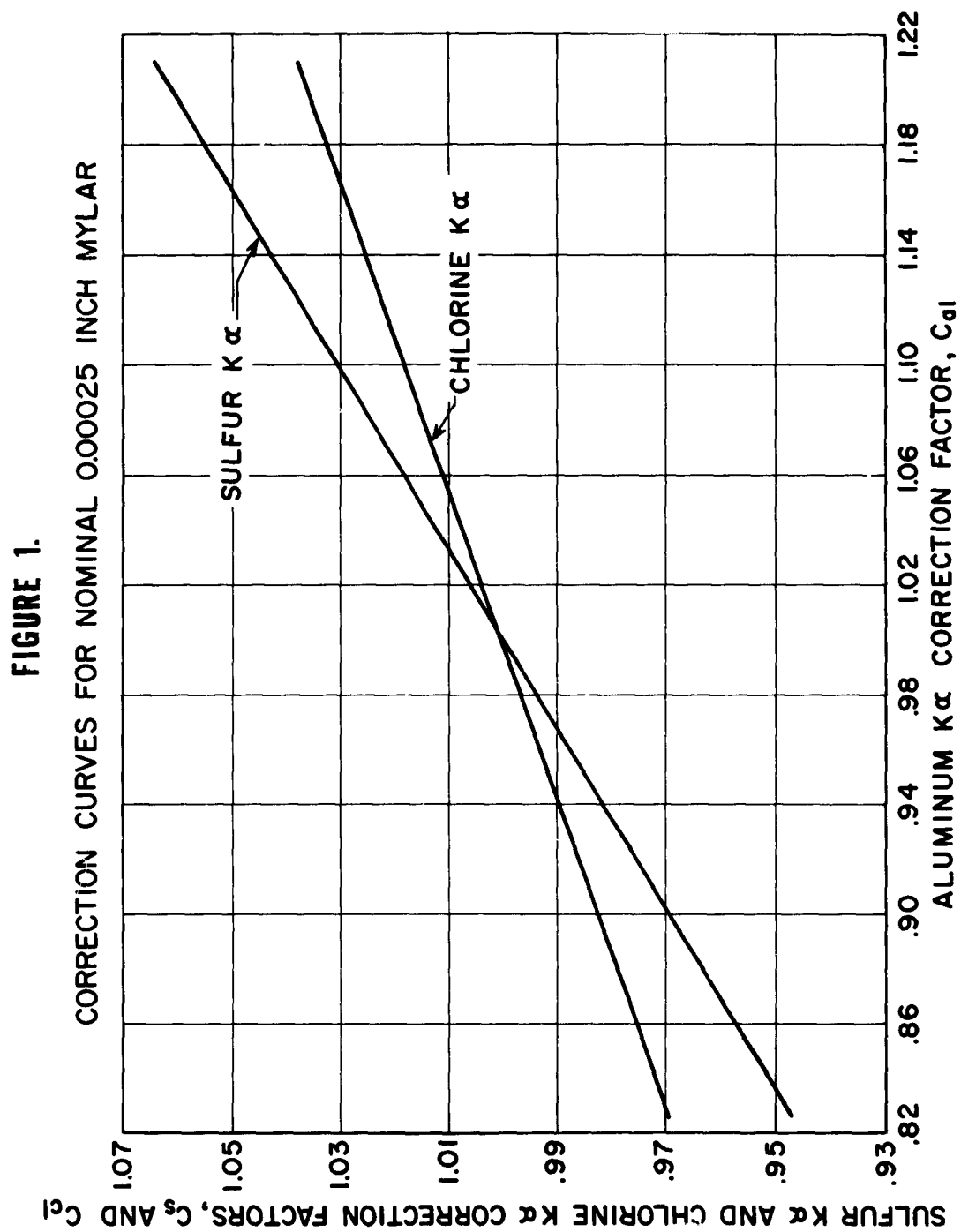
TYPICAL DATA FOR PREPARATION OF MYLAR CORRECTION CURVE

Mylar Sample	Aluminum K α		Chlorine K α	
	Seconds for 128,000 Counts	Ratio	Seconds for 1,024,000 Counts	Ratio
1	47.55	1.051	54.65	1.009
2	45.25	0.952	54.15	0.991
3	45.80	1.084	53.05	1.016
4	42.25	0.922	52.20	0.984
5	48.95	1.112	48.95	1.019
6	44.00	0.899	48.05	0.982
7	46.25	1.073	49.45	1.013
8	43.10	0.932	48.80	0.987
9	45.40	1.006	52.10	1.001
10	45.15	0.994	52.05	0.999

TABLE IV

APPLICATION OF MYLAR CORRECTION FACTORS

Sample	Aluminum Standard, Seconds	Chlorine K α			Sulfur K α			Aluminum K α		
		Correction Factor	Analysis, Seconds	Corrected Analysis, Seconds	Correction Factor	Analysis, Seconds	Corrected Analysis, Seconds	Correction Factor	Analysis, Seconds	Corrected Analysis, Seconds
Reference Standard	41.7 - 41.4	-	23.7	23.70	-	38.8	38.80	-	60.9	60.90
1	45.2 - 45.4	0.9858	40.5	39.92	0.9743	46.7	45.50	0.9172	37.3	34.21
2	44.1 - 43.8	0.9908	39.6	39.24	0.9829	47.3	46.49	0.9454	36.0	34.03
3	43.3 - 43.0	0.9939	40.0	39.76	0.9883	46.3	45.76	0.9629	34.4	33.12
Reference Standard	41.8 - 41.7	-	23.7	23.70	-	39.2	39.20	-	61.1	61.10
4	45.7 - 45.7	0.9851	40.0	39.40	0.9732	48.0	46.71	0.9136	37.8	34.53
5	46.7 - 46.7	0.9816	40.5	39.75	0.9672	48.5	46.91	0.8940	38.0	33.97
6	47.6 - 47.5	0.9788	40.4	39.54	0.9622	47.6	45.80	0.8780	38.6	33.89
Analytical Ratio				0.598			0.844			1.80
Counts Collected	128,000		512,000			16,000			16,000	



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E. W. JAMES

Director, Propulsion Laboratory

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